# Performance of BL35XU for High Resolution Inelastic X-ray Scattering

Investigation of sample dynamics at meV energy scales and Å length scales is now possible at beamline **BL35XU** [1] using inelastic X-ray scattering (IXS). IXS offers some unique advantages as compared to neutron scattering techniques, but comes at the expense of an extremely technically demanding setup. The primary advantages of IXS appear in measurements of disordered materials and small samples. More precisely, kinematic restrictions in neutron scattering can make it difficult to work at small momentum transfers, which is a crucial region for observing collective modes in non-crystalline materials [2]. In addition, typical neutron beam sizes are ~ cm$^2$, so that comparably large samples are needed. This may be compared to an X-ray beam from a 3rd generation synchrotron source that can easily be focused to a spot of ~ 100 μm or less in diameter. This is a great advantage for the investigation of materials that are difficult to prepare, or for the use of extreme sample environments (high pressure and/or high temperature).

The technical difficulty of IXS stems from the extremely high energy resolution required. Whereas meV resolved measurements using neutrons with ~ 20-meV energy require resolutions of $\Delta E/E$ ~ %, with X-rays of energy a few tens of keV, the required resolution jumps some five orders of magnitude to $\Delta E/E \sim 10^{-7}$, leading to a commensurate increase in difficulty. Despite this, BL35XU has gone from the very first tests of the spectrometer using a single analyzer in May and June of 2001 to a working setup with four analyzer crystals having 2-meV resolution in the space of five months, (including the two and a half month summer shutdown), very much faster than any comparable facility. Since the first user experiments in October 2001, steady progress has improved the resolution to between 1.5 and 1.8 meV (depending on the analyzer crystal) with as much as $5\times10^9$ photons/s in a 0.8-meV bandwidth and a ϕ 75-μm spot at the sample.

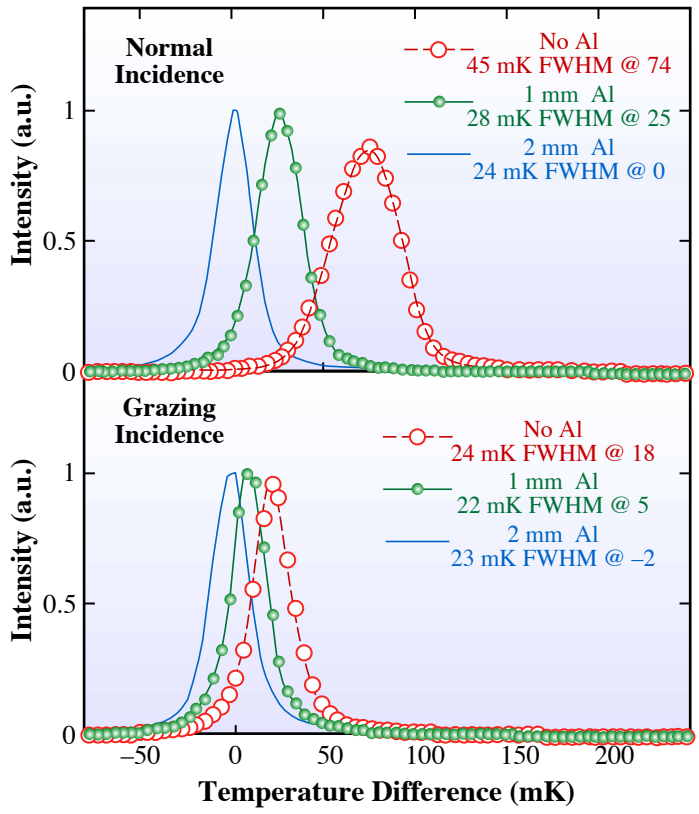


*Fig. 1. Energy resolution for different power loads on the backscattering monochromator at 21.75 keV. A flat crystal (at fixed temperature) was used to analyze the beam reflected during a temperature scan of the monochromator. Each mm of aluminum reduces the incident power by a factor of 2. The grazing incidence geometry removes all of the broadening caused by the ~100 mW of beam (no Al). It also reduces the shift that occurs because the temperature sensor is not exactly at the beam spot. (Note: the relevant scale factor is about 18 mK/meV at this X-ray energy).*

Here, we briefly discuss some of the instrumentation that has made this possible.

Two essential components in the operation of the spectrometer are the high resolution backscattering monochromator and the analyzer crystals. The monochromator, having to accommodate only the divergence of the undulator beam (some $15 \times 40$ μrad$^2$, or less) is the easier one, but, even so, it provided one notable surprise (beyond the expected difficulty of dealing with mK temperature control). In particular, it was found that the beam from the high heat load (Si (111)) monochromator, ~100 mW of power, caused local heating and distortion of the backscattering crystal over the beam spot. Thus, it was necessary to reduce the power density onto the backscattering monochromator in order to achieve resolution better than 2 meV. This was done by replacing the normal incidence backscattering monochromator with a grazing incidence version: working at a grazing angle of some 2.5 degrees reduced the power density by a factor of 20, and removed nearly all the effects of the heat load, as is evident in Fig. 1.

The analyzer crystals are more difficult, as they must accommodate a solid angle of $\sim 10 \times 10$ mrad$^2$, some five orders of magnitude more than the monochromator. Thus there has been an ongoing R&D program with NEC Fundamental Research Laboratory over the last four years to achieve good quality crystals. We have been following a prescription similar to that of [3]: first a flat wafer is cross-cut to produce many free-standing crystallites with a thin common back-wall; then these crystallites are bonded to a substrate of appropriate curvature; and finally the common back wall is removed, leading to many independent and, importantly, unstrained crystallites with the correct orientation. This process remains a bit of an art, but slow and mostly steady progress has led to a set of four crystals giving resolutions of 1.5, 1.6, 1.6 of 1.8 meV (operating at a 10-m radius, without any limiting apertures) using the Si (11 11 11) reflection at 21.75 keV. Presently, a 60- or 70-μm thick saw blade is used to cut 2.9 mm into a flat wafer on a 750 μm pitch. The crystallites are then bonded to a spherically curved substrate using a high temperature gold diffusion bond, and the back-wall is removed, leaving about 15000 independent crystallites with the appropriate geometry (rms deviation from the 9.8-m radius of the substrate is ~15 μrad, or better). Notably, after all etching, the active area of the crystallites is 60 to 65% of the substrate area, which is rather good. Figure 2 shows one of the analyzer crystals, while the Table I gives detailed

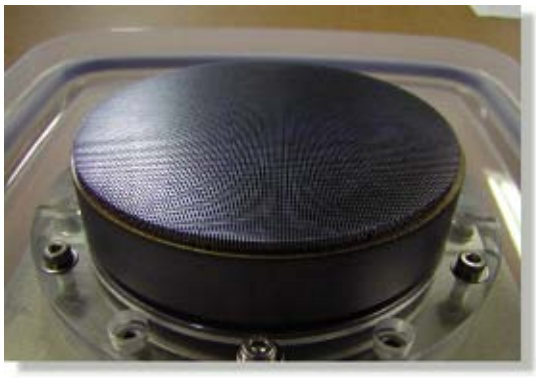

*Fig. 2. 10-cm diameter analyzer crystal. Resolutions are presented in the Table I.*

**Table I.** *Resolution at various silicon orders with the best analyzer crystal.*

| Energy | Si (n n n) | FWHM (meV) | FWM/10 (meV) | FWM/100 (meV) | Rel. Flux |
|---|---|---|---|---|---|
| 15.81 | (8 8 8) | 6.1 | 15.3 | 47 | 8 |
| 17.79 | (9 9 9) | 3.1 | 8.3 | 29 | 2 |
| 21.75 | (11 11 11) | 1.5 | 4.2 | 16 | 1 |

FWHM = Full Width at Half Maximum
FWM/X = Full Width at Maximum over X

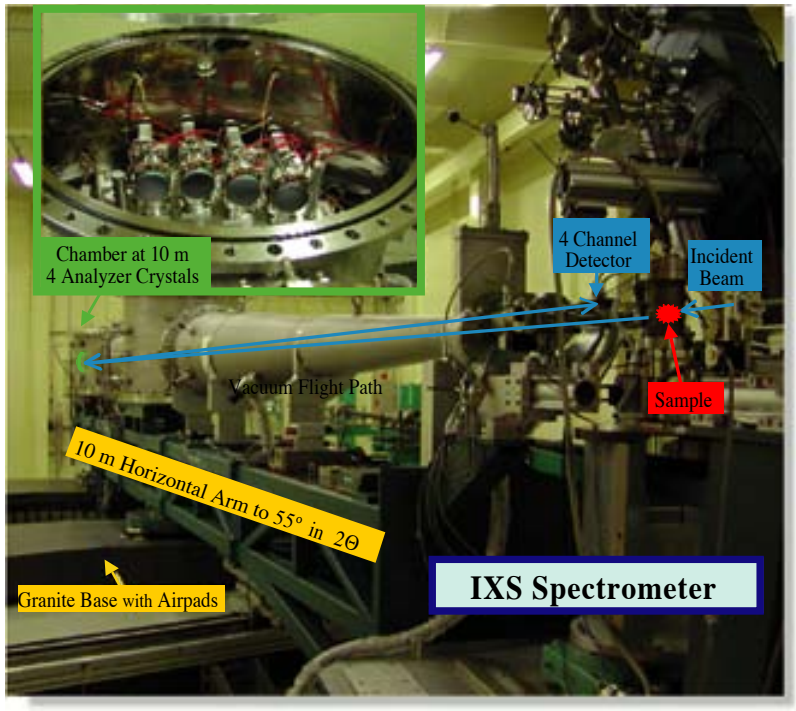


*Fig. 3. The horizontal arm of the IXS spectrometer. Focused, highly monochromatic beam is incident from the right and is scattered by the sample into a set of four analyzer crystals that focus it into four detectors, allowing simultaneous measurement of four momentum transfers.*

parameters. We are presently working to improve all crystals to the same 1.5-meV level of resolution.

The IXS spectrometer is shown in the final figure (Fig. 3). In the 19 months since first beam from a single analyzer crystal, and the 14 months since opening for user operation, the spectrometer has been used by many user groups to investigate a variety of liquid and solid materials, including several types of molten metals, mercury near the liquid-gas critical point (so both at high temperature and high pressure), and many crystalline materials, including several types of superconductors and one quasi-crystal.

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**References**

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[2] F. Sette *et al.*, Science **280** (1998) 1550; S. Hosokawa *et al.*, in this issue of SPring-8 Research Frontiers.
[3] C. Masciovecchio *et al.*, Nucl. Instrum. and Meth. B **111** (1996) 181.